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ICE PLUGGING OF PIPES
USING LIQUID NITROGEN

by

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USING LIQUID NOTROGEN

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ABSTRACT

This report presents a study on the ice plugging of pipe using liquid nitrogen, and is based on a literature review and on discussions with individuals who use the technique. Emphasis is placed on ferritic alloys, primarily carbon steels, in pipe sizes up to 60 cm in diameter and on austenitic stainless steels in pipe sizes up to 30 cm in diameter.

A limited number of case histories is used to outline operating experience. Also included are recommendations from users of the technology - recommendations intended to highlight the hazards when the technique is applied to ferritic alloys and the means by which these hazards can be partially controlled.

RÉSUMÉ

Le rapport présente une étude des obturations faites sur des canalisations par congélation d'azote liquide, et il est fondé sur un examen de la documentation et sur des discussions tenues avec des personnes qui utilisent cette technique. L'accent est mis sur les alliages ferritiques, principalement l'acier au carbone, pour les canalisations de taille allant jusqu'à 60 cm de diamètre, et l'acier inoxydable austénitique pour les canalisations de taille allant jusqu'à 30 cm de diamètre.

Un nombre restreint de cas témoins sert à expliquer l'expérience en matière de fonctionnement. Il y a aussi des recommandations des utilisateurs de cette technique, qui visent à expliquer les dangers qui existent lorsque cette technique est appliquée aux alliages ferritiques et les moyens qui permettent de contrôler partiellement ces dangers.

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ICE PLUGGING OF PIPES
USING LIQUID NITROGEN

1. SCOPE OF WORK

Information on the freeze plugging of piping using liquid nitrogen was compiled through a literature review and discussions with individuals who are using the technique. Particular emphasis was placed on ferritic steels in piping up to and including 600 mm (24 in.) in diameter and on austenitic stainless steels in piping up to and including 300 mm (12 in.) in diameter.

The purposes of the review were:

- (a) to identify the extent to which freeze plugging of ferritic and stainless steel piping is practiced,
- (b) to identify failures that occurred during, or as a consequence of, freeze plugging, and
- (c) to rank, if practical, the parameters which can be considered detrimental to the integrity of piping subjected to freeze plugging.

2. HISTORY OF ICE PLUGGING

Although ice plugging has been known for many years, initially in freezing of lines at ambient winter temperatures, its use as a testing or maintenance procedure was recommended by NASA Publications in the late 1950's to early 1960's. Subsequent Publications described the utilization of dry ice to form plugs in tubing carrying liquid coolant; blockages could be then flushed out without contaminating the system fluid with the flushing medium¹. Later, freezing techniques were adapted to the testing of piping systems, particularly buried lines, to locate leaks by isolating progressively smaller sections of the line until the leaking section was identified².

A patented freeze jacket that could be applied to piping systems was developed in the United States, primarily for use in leak-testing services. This jacket permitted much better control of the freezing operation than was possible with the crude coils, boxes and other equipment devised earlier².

Even with the freeze jacket, the majority of work still involved the leak testing of lines. However, it was obvious that the freeze plug method could be applied to lines containing few block valves. This is particularly true in nuclear facilities where the number of valves is limited due to leakage considerations. There, freeze plugging permits the formation of a positive seal, allowing maintenance on a line without completely draining the system, thereby saving considerable time and inventory.

Ice plugging was virtually ignored in the literature until the late 1970's. Then a vigorous marketing campaign, including extensive publication, took place in both the United States and Great Britain primarily by three companies, one American and two British^{3,4,5,6,7}. These papers described freeze jackets used to produce ice plugs in piping up to 760 mm (30 in.) in diameter. Publication, particularly in Great Britain, ceased abruptly at the end of 1982. It was rumoured that the United States company had charged one of the British companies with patent infringement and an out-of-court settlement was reached.

At the same time, work on forming ice plugs in piping systems was underway elsewhere. In Germany, particularly in the nuclear industry, a number of papers appeared throughout the 1970's and even more were published in 1984 and 1985. Similarly, investigations in France led to the development of an ice plug technique for use in that country's thermal and nuclear power stations^{8,9,10}. In addition, recent publications indicate that the technique is being studied and, presumably, applied in Japan¹¹, India and China.

In Canada, freeze plugging has been used for many years by individual companies to permit maintenance on cooling water lines and other non-hazardous systems. Few records were kept on the use of the technique since it was considered to be a basic maintenance procedure.

Freezing certainly took place in the early years of chemical plants but the first documented application occurred in 1962 or 1963 in Quebec¹². It was used sporadically at other plants as details of the technique circulated orally.

In the early 1970's a group was established in Sarnia, Ontario to examine ice plugging, evaluate its effects on piping material, and establish a procedure for the technique in the petrochemical and refining industries¹³. A number of experiments and field trials were conducted over a two-year period, culminating in the issuance of a procedure to the member companies for use of the freezing technique on water and other non-hazardous systems. The procedure, which was never used, was lost.

Canadian interest in ice plugging was revived in the late 1970's during a marketing campaign that was launched in the United States. Many companies tried the system at least once, with some opting to include it as a maintenance tool and others rejecting it, citing a fear of brittle fracture of carbon-steel piping components. The power generation industry, particularly nuclear facilities, appears to have been a major user of the technique in Canada as well as elsewhere.

Reluctance to use the technique appears to be based primarily on concerns about brittle fracture in carbon-steel components because freezing temperatures are considerably lower than the NIL-DUCTILITY TEMPERATURES of the material. In addition, accidents, either actual or rumoured, were cited as confirmation of the degradation of piping materials due to freezing. A comprehensive testing program completed in 1982 attempted to determine the validity of the concerns of brittle fracture during freezing operations¹⁴. While the program demonstrated that ice plugging was a viable pipe isolation technique, it recommended additional testing to examine fracture tendencies, during pipe freezing, of ferritic alloys containing various defects.

3. PERCEIVED PROBLEMS WITH ICE PLUGGING

There are many methods for blocking piping systems or pipelines to permit hydrostatic testing or maintenance operations. Usually involving the insertion of mechanical devices to block the flow, they are particularly applicable to lines containing block valves or other openings allowing access to the pipe. The ice plugging method appears more suitable in situations in which either block valves are not present or, extremely long pipe runs exist between access points.

Ice plugging involves the formation of a blockage in a line by freezing liquids, most often water, in the pipe by the application of a coolant in a heat exchanger. The coolant reduces the temperature of the liquid until an ice plug is formed. To isolate the water from the product in a pipeline, the water is moved between two batch pigs¹⁵. Consequently, this water slug may be moved up or down the lines, as required, to form ice plugs at other locations.

Once an ice plug has been formed, an isolated section of the line may be tested hydrostatically or repairs made - the ice plug acting as a seal against the contents of the line.

Although ice plugging appears to be relatively fast and certainly economical, a number of technical and practical concerns have been raised. These concerns tend to restrict the use of ice plugging, particularly with ferritic alloys which are subject to a ductile/brittle transition in fracture mode with decreases in temperature. Among the concerns most frequently cited are the following:

- (a) the potential for brittle fracture of carbon-manganese steel piping material at the low temperatures required to effect freezing; the propensity for fracture is increased by the presence of small defects in steel, defects that are innocuous at ambient temperature but may be significant at lower temperatures,
- (b) the permanent deformation of piping by an ice plug, probably leading to rupture of the pipe due to ice pressure, or the introduction of stresses of sufficient magnitude to cause stress corrosion cracking under operating conditions,
- (c) the possible slippage of the ice plug (thereby releasing the contents of the pipe line),
- (d) the potential for damage to components downstream by the slipping ice plug,
- (e) the possibility that the alloy structure might be altered by the low temperature of the liquid nitrogen (thereby changing the properties of the piping material),
- (f) the lack of data and documented case histories on use of the technique in open literature,

- (g) the reluctance of many companies to acknowledge use of the technique and the rumours of accidents that are neither confirmed nor denied.

3.1 Brittle Fracture

In ferritic alloys, a change from a ductile to brittle fracture pattern occurs with lowering of temperature. This transition occurs over a narrow temperature range and usually is presented graphically as impact energy versus temperature^{11,14}. The presence of sharp notches, abrupt changes in section thickness, and weld defects^{13,16,17,18} may contribute to the propagation of cracks, resulting in rupture of the pipe.

In addition, manipulation of the pipe, impacts on it, or other sources of inducing high stresses while at very low temperatures, may produce fracture¹⁸.

These considerations were addressed when the properties of a carbon manganese steel pipe that had been subjected to a single freeze test were examined before and after exposure to liquid nitrogen. No significant changes were detected, as a result of the exposure, in strength, toughness or microstructure¹⁹.

The above trials were conducted on seamless pipe so the influence of weld defects was not examined. Earlier trials in Sarnia had suggested that weld defects were of considerable concern in producing fracture¹³. However, some of the failures in the Sarnia experiments may have been the result of crude freezing techniques. Extensive testing at Battelle Columbus Laboratories revealed that precracked samples did not fail under freeze conditions, even though the cracks were in the most highly stressed areas and fracture was predicted by conventional theory^{14,20}.

It would seem that brittle fracture can be avoided in commercial alloys, by exercising strict control over the freezing operation¹⁴.

3.2 Deformation

The second concern, deformation of the piping material attributed to ice formation, arises from observations of failures in piping systems under winter conditions. Here, rupture is accompanied always by swelling of the pipe and it had been assumed that ice pressure was responsible. It is now generally accepted that increasing hydraulic pressure in regions of unfrozen water trapped by ever expanding ice, causes the fracture^{14,21,22}. During ice plugging this problem is controlled by venting pressure as the freeze progresses.

3.3 Plug Slippage

Slippage of the ice plug is a concern that was recognized early in freeze operations. This event was most often attributed to failure of the refrigerant supply or to premature removal of the freeze jacket²³. On the other hand, it has been demonstrated that when using liquid nitrogen, the cooled

pipe attains an "hour-glass" configuration, forcing the ice into a conical wedge shape and locking it in place. Thus through the use of appropriate procedures and the adoption of certain precautions, slippage of the plug can be avoided.

3.4 Changes in Microstructure

Pipe freezing using liquid nitrogen should not produce any changes in microstructure of low carbon-manganese steel pipes^{16,24}. In steels where a transformation of retained austenite to martensite may occur, a slight decrease in toughness may result. However, the transformation is so slow that many days' exposure to liquid nitrogen temperatures would be required to effect a change in properties. Most commercial freezes result in a few hours' exposure to low temperatures. Concern about transformation may be very real for repeated freezes at the same location, or for freezes on large diameter piping as long periods of exposure to low temperatures are required to produce an ice plug.

3.5 Lack of Documentation

The final concern - lack of data and case histories, and company reluctance to discuss the technique - is certainly justified. There is relatively little information in the open literature and most of what is available has been generated by those selling a commercial freezing service. There are exceptions: discussion of a procedure developed to test waste lines²⁵, studies conducted at Battelle Columbus Laboratories¹⁴, studies by Electricité de France^{8,9,10}, and a proposed project in the United Kingdom²³. These studies address safety of the procedure, effect on material properties, stresses generated, and general considerations to prevent failure of piping components.

Until the Battelle study, there was no systematic analysis of the effects of defects in the piping material on brittle fracture in ferritic alloys under freeze conditions. Applications to carbon steel alloys were based on a very limited study conducted on a small-diameter seamless pipe containing no reported defects¹⁹.

The case histories that do appear in the literature tend to be extremely brief indicating where and when a freeze was performed. They are reported by those performing the freeze, not by plant personnel where the freeze took place. Potential or actual problems are not reported.

To supplement the limited published information, individuals in plants throughout North America and the United Kingdom were contacted. Reaction to our request varied; some people discussed their experiences readily but many others refused to discuss any experience with ice plugging.

4. METHODS OF FREEZING

Although the use of liquid nitrogen to effect freezing is the focus of this study, there are other media such as carbon dioxide or chlorinated hydrocarbons that may be employed; the criterion being that a heat transfer medium must reach a sufficiently low temperature to cause freezing in other liquids, principally water³⁶. Many of these processes are known and marketed by trade names.

Such processes use a sleeve, jacket or freezer head to contain the freezing medium. The jacket is applied over the area to be frozen and the freezing medium is introduced. The appearance of frost on the piping outside the jacket area is considered a sign that the plug has formed. Jackets or sleeves may be of several types:

- (a) portable containers which may involve a split cylindrical or rectangular device that can be clamped in place,
- (b) fixed cylindrical jackets which are applied to piping during construction and then sealed by the application of rubberized compounds that are injected prior to the coolant,
- (c) permanent cylindrical jackets welded to the piping,
- (d) internal freezing devices inserted into a pipe through a valve or other opening, and
- (e) freezer heads that can be clamped around the pipe.

The jacket materials are many and varied depending on the extent of the freeze.

Aluminum and austenitic stainless steels are the most popular metallics employed, although copper jacketing has been used on occasion. Fibreglass jackets have been patented and are used commercially¹⁵. These jackets are preformed in standard pipe diameters up to 1120 mm (48 in.).

For small diameter piping, jackets may be made from wood, styrofoam or any other compound suitable for containing the freezing medium. It is not unusual to find rough styrofoam boxes made by those attempting a freeze.

Piping freezing techniques and equipment are marketed under a variety of trade names. Table 1 outlines some trade names and freezing methods that were identified in published literature.

PIPE FREEZING PROCESSES

TRADE NAME	SUPPLIER	FREEZING MEDIUM	TYPE OF SLEEVE	PIPE SIZE
Arctic Spray	Eldy Ind.	CCl_2F_2	polyurethane foam	25 mm (1 in.)
Cardice	Distillers Co. Ltd. UK.	CO_2 (Dry Ice)	insulated boxes	400 mm (16 in.)
Cryo Speed	BOC Ltd.	Liq. N_2	double walled cylinder	750 mm (30 in.)
Drikold	ICI Ltd.	CO_2 (Dry Ice)	insulated boxes	400 mm (16 in.)
Ice-O-Later	Freeze Technology Int.	Liq. N_2	double walled cylinder	1200 mm (48 in.)
Jet Freezer	Distillers Co. Ltd. UK	Liq. CO_2	nylon	100 mm (4 in.)
Qwik Freezer	COB Industries Inc.	CCl_2F_2	freezer heads	100 mm (4 in.)
ROHR Frost	Consort Rothenburger Ltd.	CCl_2F_2^*	freezer heads	54 mm (2 in.)
Viraglace	Virax UK Ltd.	CCl_2F_2	aluminum	50 mm (2 in.)

* Liquid nitrogen is now used as well. Details are available in German.

Information obtained from published literature and may not reflect current operations or capabilities.

5. EXPERIENCE WITH ICE PLUGGING

Based on published information, the extent of ice plugging to effect a seal in piping systems for maintenance purposes is limited. However, the practice seems more widespread than is indicated in the literature.

The origin of the method is unknown. Probably, many individuals realized independently the potential for freeze plugging based on their observations of accidental freeze sealing of lines under ambient winter conditions in northern climates. The earliest reference to ice plugging found in the literature appears to be in 1968³⁷, but undoubtedly, there are earlier publications. In any case, references to the technique appear sporadically until 1976 when a number of companies, primarily in the United Kingdom, began to publish articles extolling the virtues of the process^{3,4,5,6,7,28,29,35,38}. Most of these articles contain little technical detail of the process, simply reporting abbreviated case histories. In some instances, the announcements were premature, as later work indicates that reported freezes were never completed^{28,35}.

Publications on the freeze technique appear frequently in literature from 1976 through to 1985. For the most part, the articles outline the advantages of the technique without addressing any of its potential problems. Exceptions include a proposed study in the United Kingdom²³, work in France^{8,9,10} and an extensive study at Battelle Columbus Laboratories¹⁴. These works address the effect of the process on the metallurgy of the piping material and the potential for brittle fracture in carbon steels. Similarly, the Sarnia study¹³ was more concerned with possible material problems than with the success of the process.

English abstracts of German and Japanese publications indicate an increased use of the technique in those countries. Liquid nitrogen appears to be replacing other refrigerants in the processes and patents are being applied to certain design features of cooling jackets and/or refrigerant heads.

Publications on the freeze technique and advertising of the service appear to end in the United Kingdom in late 1982. Although information was readily available prior to 1983, there are no current references to work being done nor is the service offered in major British trade catalogues.

Conversations with individuals in some major corporations in the United Kingdom indicated that while the service is still available, its use is not as widespread as it once was. The reason appears to be at least one serious accident and possibly a number of minor accidents. However, it must be emphasized that this information has not been confirmed in writing.

In France, the government has withdrawn its approval of the use of ice plugging for maintenance purposes on primary circuits in nuclear facilities³⁹. This action was taken despite studies that have shown that the process does not affect piping materials.

It appears that while the use of ice plugging is rising in North America, particularly in nuclear facilities, it is being restricted in other industries. This restriction reflects the cyclical nature of ice plugging and

other hot tapping procedures in that their use expands until accidents or adverse publicity are encountered. For a few years use is reduced and then gradually expands again.

Ice plugging was first used as a leak locating procedure². The approach was to divide lines into sections by forming ice plugs, hydrostatically testing the sections and eventually isolating the leak by decreasing the length of the section. This would limit costly excavation of an entire line to locate a leak.

Cryogenic Engineering is a well developed discipline which is utilized in the production of liquified gases and other systems requiring refrigeration. As with any industrial process, safety is of prime concern.

It has been established that brittle fracture may occur in alloys used at cryogenic temperatures if:

- (a) a stress riser such as a notch, crack, or lamination is present,
- (b) stress exceeds the yield stress, or
- (c) the temperature is below the NIL-DUCTILITY Transition level^{17,18}.

In addition, the ASME Code Section VIII - Division I established design stresses for cryogenic service as those that exist at 38°C (100°F)¹⁷. However, special tests may be required for material that is to be subjected to low temperatures on an occasional basis. In France, for example, non-destructive evaluation is performed on the pipe before and after the freezing operation³⁹.

Studies in Europe and North America have concluded that carbon steel piping materials may be frozen without damage to the structure^{8,9,10,19}. Although many of these tests are limited to a few samples, they do demonstrate that no structural changes occur during the cooling and heating cycle.

More extensive studies have sought to examine the effect of notches under freeze conditions. Laboratory trials on piping materials have shown that the presence of a notch in the freeze area has little or no effect on the material integrity¹⁴, possibly due to a number of factors, including the conservative nature of an analytical model for brittle behaviour and an increase in tensile strength with decrease in temperature.

In contrast, field trials have revealed that the presence of 1.5 to 2 mm deep notches in the freeze area, particularly when associated with welds, is sufficient to cause fracture under freezing conditions¹³. These notches may well be overlooked by standard Non-Destructive Testing techniques. However, it must be emphasized that these trials were conducted with crude homemade freezing devices which may have contributed to the failures.

Following the success of freeze block testing on buried plant piping systems, pipelines, and other transmission lines, its use was expanded to above-ground systems where it was applied as a maintenance tool, particularly in the power industry⁴⁰. Here, limited valving prevented the isolation

of systems and freeze plugging could be used locally, to eliminate the draining of an entire tube bank for maintenance.

The practice of freeze plugging in the power industry seems widespread. A recent survey of nuclear power facilities revealed that most respondents were performing their own freeze plugging on piping up to 150 to 200 mm (6 to 8 in.) in diameter²⁴. Freezing was applied, on a limited basis, to piping of larger diameter but a commercial freezing company was contracted to do the work.

In Canada, freeze plugging has been used at least since 1963 when the technique was employed at Shawinigan Chemicals¹². However, as there are no records of the early years, details of freeze applications at various plants are based on personal reminiscences of those involved.

Most freeze applications involved 150 to 200 mm (6 to 8 in.) diameter austenitic stainless steel lines. Freezing was accomplished using a number of compounds such as carbon dioxide, ethylene, ammonia, and liquid nitrogen. A jacket, generally of stainless steel but sometimes of wood, was built around the pipe and the coolant applied. Once ice formed on the pipe outside the jacket, the plug was considered to be solid. Stress measurements were not performed on the pipe and the structure was not examined for damage unless failure occurred.

Freezing under these conditions resulted in some pipe deformation¹² but this presented no problem during subsequent operation.

An unconfirmed report attributed the failure of a stainless steel line in 1969 in Varennes, Quebec²⁶ to expansion of the line during the freezing operation. The line experienced a longitudinal crack in the area where freezing had occurred. Microscopic examination identified chloride stress corrosion cracking as the cause of failure. The stress in the line was attributed to swelling caused by the ice plug.

In 1968 and 1969, a number of carbon steel lines associated with a cooling tower were frozen to permit maintenance. Later these lines experienced a considerable increase in the rate of corrosion in the areas that had been frozen so the technique was abandoned²⁶.

Field trials conducted by Dow and Polymer (now Polysar) tested various carbon steel, austenitic stainless steel and high nickel alloy lines and the metal structures were examined microscopically. Failures were experienced, particularly in carbon steel, welded sections. The failures were brittle in nature and were associated with weld defects such as incomplete penetration, incomplete fusion, misalignment, cracks and, to a lesser extent, porosity. Swelling of lines was detected but stress corrosion was not evident although increases in corrosion rates were noted on some carbon steel lines. A procedure for freezing was developed but the technique was restricted to water or other non-hazardous systems.

With the exception of the Canadian Nuclear Industry, ice plugging appears to have been used at many plants only once (although there are a few facilities where the technique has been used several times). Most freezes took place from 1978 to 1982 and involved a single application to a 150 to 200 mm (6 to 8 in.) diameter carbon steel line. Reaction to the process has been mixed.

Many facilities indicated that the freeze technique produced the desired blockage in the line. Following the freeze, the lines were returned to service and operated, in most cases, for several years without incident.

Other facilities expressed concern over the effects the freezing might have on piping materials and indicated that there was insufficient technical data to support the claim of no damage. They complained that there was no method of positively establishing that the ice plug had formed and that the appearance of ice on the pipe could be misleading as they had many operating ice-covered lines through which flow continued.

Major industries in Canada appear to be divided equally on the use or prohibition of ice plugging. Those supporting its use cite economy and ease of application as positive features. However, even these facilities are exercising extreme caution following rumours of failures and possible insurance problems. In addition, some facilities have performed their own freezing on small-diameter lines and admit to pipe fractures.

The opponents of freeze plugging state that the process is dangerous when applied to carbon steel lines and that failure could result during the cooling and heating operation. In many cases, they tried freezing years ago and experienced difficulties with plugs breaking loose, plugs not completely formed, and fracture of pipe.

As well, failure has been experienced in piping systems which have frozen under winter conditions. Although such fracture is probably caused by the build-up of pressure (bled off during an intentional freezing operation), an error in venting could lead to failure.

The procedure remains popular with certain segments of industry, but its use appears to have been severely restricted since 1982 and even its most ardent supporters, outside the nuclear industry, are advising extreme caution. They suggest the technique should be used only in an emergency and then only by those fully experienced in its application.

5.1 Survey of Canadian Industry

As a measure of the extent of the use of ice plugging in Canada, thirty-four major Canadian companies were contacted. These companies represent all segments of the chemical, petrochemical, refining, and gas industries and most have facilities in more than one location across Canada.

Seventeen of the thirty-four companies replied that they had used a freeze plug technique on at least one occasion for leak testing or maintenance

purposes. The procedure was carried out on piping 150 to 200 mm (6 to 8 in.) in diameter. Only three of these companies indicated they had used the technique more than once.

Most of the freezes occurred from 1978 through to 1982 and many involved only a demonstration of the technique. In most cases the freezing was carried out by a commercial freeze company. Metallurgical studies were not conducted on the piping material either before or after the freeze.

Twelve of these seventeen companies indicated they would use ice plugging again on an emergency basis but would not consider it as a regular maintenance procedure. All stated that extreme caution must be exercised to prevent fracture of the pipe but as long as proper procedures were followed no problems should be encountered.

The remaining five stated they would not use ice plugging in their facilities as the possibility of brittle fracture in welded and corroded pipe was very real. All quoted instances of fractured pipe and vessels under winter conditions and some referred to fractures during ice plugging operations. It was unclear, however, whether these fractures occurred during a commercial freeze or during attempts with homemade equipment.

The other seventeen companies replied that they had not used the ice plug technique nor had they any intention of doing so. They indicated that there was insufficient data to support claims that the freezing did not affect piping materials. As well they believed brittle fracture in ferritic alloys could be a problem.

A similar survey was attempted in the United States but received fewer responses. Twelve companies replied, indicating that each had used the technique at least once, again only on piping up to 200 mm (8 in.) in diameter. A few problems were encountered but with only one exception⁴³, details were not available.

In Canada, the nuclear industry continues to be the most frequent user of the ice plugging technique. However, it has been used occasionally in chemical, petrochemical, and gas plants as well as in refineries. Claims that it has been used in the pulp and paper industry could not be confirmed.

Based on our survey, it would seem that the frequent or regular use of the technique is not widespread in Canada nor indeed elsewhere, outside of the power industry.

6. CASE HISTORIES

The following case histories were obtained from the open literature, contacts with various companies, and with individuals who were able to recall some details of work carried out years ago. A number of companies supplied information but declined to be identified so that some data could not be documented.

6.1 1963 - Shawinigan Chemicals¹²

A 150 mm (6 in.) nominal diameter austenitic stainless steel line, probably type 304, in hydrogen service was frozen using ethylene so repairs could be made. A slug of water was frozen in the line by introducing ethylene into a stainless steel jacket built around the pipe. Some distortion was noted in the pipe but no corrosion and/or fracture problems were encountered.

6.2 1969 - Varennes, Quebec²⁶

A 150 mm (6 in.) diameter line of type 316 stainless steel was frozen using liquid nitrogen. The line displayed some swelling and then ruptured a few days after being put back in service. A longitudinal crack was found in the area that had been frozen.

Microscopic examination indicated that chloride stress corrosion cracking was the cause of failure. The stresses were attributed to swelling of the line during the freezing operation.

6.3 1968-69 - Varennes, Quebec²⁶

Several carbon steel lines from a cooling tower were frozen using carbon dioxide. Following their return to service, the lines experienced an unexplained, dramatic increase in corrosion rates in the areas that were frozen so use of the freezing technique was abandoned.

6.4 1971-73 - Sarnia, Ontario¹³

A committee with representatives from Dow Chemical, Polymer, Cabot Carbon, Shell Canada, and Ethyl Corporation was formed to study ice plugging and to produce a procedure for its use. Trials were conducted at Dow and Polymer under the direction of Messrs. Futch and Twigg who also carried out metallographic analyses on the piping alloys.

Alloys used in the experiments conducted in field pipe loops were:

Carbon Steels - A53 Grade B
 - A106 Grade B

The material tested was up to 400 mm (16 in.) in diameter, schedule 40. It was all seamless but, for test purposes, some sections were split and longitudinal welds were made. Weld defects were intentionally produced in some areas to test their response to freezing. In addition, some old, corroded pipe was tested as well.

Stainless Steels - A312 types 304 and 316

Again, material up to 400 mm (16 in.) in diameter and schedule 40 was tested. Piping over 150 mm (6 in.) in diameter was welded. In addition, simulated welds were made by splitting pipe and rewelding so that weld defects could be introduced.

Higher Nickel Alloys - Incoloy 800
- Alloy 20 Cb3

Both alloys were tested in piping up to 200 mm (8 in.) in diameter.

Results of the Tests

Crude boxes were built around the areas to be tested to act as freezer chests. In some instances stainless steel tubing was wrapped around the piping and the cooling medium circulated through it.

Various cooling media, including ammonia, ethylene, carbon dioxide, dry ice and brine, and liquid nitrogen, were used. The best freezing results were obtained with the liquid nitrogen; however, the majority of the piping fractures produced during testing occurred when liquid nitrogen was the coolant. Since liquid nitrogen would produce lower temperatures than the other media, the failures could be attributed to the greater thermal gradients.

Difficulty was experienced in obtaining a plug above 200 mm (8 in.) pipe diameter, particularly using ammonia and the dry ice and brine system. Melting plugs were a very real problem and slippage was experienced under pressure tests.

Liquid nitrogen produced the best results in all tests. Generally, its plugs did not slip and untimely melting was not experienced.

Freezing of hydrocarbons presented many problems including fires generated during welding trials when plugs melted.

Fracture occurred in many of the tests on carbon steel. Invariably, these fractures were associated with weld defects, particularly those present in longitudinal welds. Cracks, incomplete penetration, incomplete fusion, misalignment and porosity, in decreasing order, were found to be the chief causes of fracture. Undercut did not result in fracture but a corrosion groove alongside the weld did, particularly on longitudinal welds.

Increased corrosion on areas that were frozen was found in A53 material but not in A106. It was suggested the accelerated corrosion was due to a subtle change in surface chemistry as a result of the freezing, possibly oxide removal, making one area anodic to the rest of the pipe.

Austenitic stainless steels and the higher nickel alloys did not fracture under test, although some swelling was observed. One section of type 304 stainless steel pipe did show a series of longitudinal, shallow, parallel cracks in the area that was frozen. Given sufficient time, these might have propagated to failure.

A procedure was developed to allow the freeze technique on seamless non-hazardous service piping up to 300 mm (12 in.) in diameter using carbon dioxide, ethylene or liquid nitrogen. However, the procedure was never used and all copies have been discarded from the member companies.

6.5 1975 - Atlantic Richfield Hanford Company, Richland, Washington²⁵

A shop test was conducted on a 6 m (20 ft.) long 75 mm (3 in.) diameter, schedule 40, carbon steel pipe using dry ice and freon. Several tests were conducted to design a jacket of the proper length to obtain a plug and to determine the flow rate of water which would permit formation of ice. It was determined that the pipe must be vented as the ice plug formed.

Following the successful shop trial, field trials on a 75 mm (3 in.) diameter, schedule 10, stainless steel waste line, 240 m (800 ft.) long were conducted. Several trials were necessary to establish the correct parameters for the process but a successful hydrostatic test was obtained using the freeze block method. Subsequently, a test procedure was issued.

6.6 1976 - Natco Inc., Amarillo, Texas¹⁹

A 200 mm (8 in.) nominal pipe size schedule 40, API 5L, Grade B seamless pipe was subjected to a freeze test using liquid nitrogen to freeze water within a 750 mm (2½ ft.) long section. A hydrostatic test to a maximum pressure of 8.16 MPa (1,185 psig) with the pipe at a temperature of -146°C (-230°F) was performed.

The pipe was sent to an independent laboratory for testing following the freeze. Tensile tests, impact tests and metallographic examination revealed no alterations in properties as a result of the freeze. This test was, for many years, the basis for concluding that fracture would not occur in carbon steel pipe under ice plugging conditions.

6.7 1977 - Report Date - Permian Corp. and Diamond Shamrock³

The freeze method was used to locate leaks on a 200 mm (8 in.) and 25 mm (10 in.) diameter pipeline in Texas, jointly owned by Permian Corporation and Diamond Shamrock. The pipeline was upgraded by repairing and/or replacing leaking sections.

6.8 1978 - Test for Colonial Pipeline as Reported by Brister Inc.²⁷

A test was carried out on a 24 m (80 ft.) long, 1020 mm (40 in.) diameter, 8.73 mm (0.344 in.) wall thickness, X 60 line pipe which was capped at each end and mounted on skids.

An ice plug was established in approximately sixteen hours. Pressure was bled from both sides of the plug and one side of the plug was subjected to a hydrostatic test at 6.77 MPa (980 psig) for seventeen hours. No problems were encountered.

Metallurgical tests scheduled to be performed on the pipe were not reported.

6.9 1978 - 4" Methanol Line as Reported by BCB Pipe Freezing Ltd.⁶

A number of plugs was maintained on a 100 mm (4 in.) diameter methanol pipeline, permitting removal and replacement of a section of line.

6.10 1978 - Reports of Freezes on Oil Platforms by David Buckman, Buckman & Cranfield Press Services²⁸

Two freezes were performed on oil platforms, one on a 400 mm (16 in.) riser and the second on a 500 mm (20 in.) branch connection. Apparently both freezes were successful.

6.11 1979 - Report by BCB Pipe Freezing Services Ltd.²⁹

A test was made of a 600 mm (24 in.) diameter concrete-lined steel pipe. A plug was tested at 1.1 MPa (225 psig). No problems were noted.

6.12 1979 - Tokyo Electric Power Company Ltd.¹¹

A freeze chamber using liquid nitrogen was developed to stop the flow of oil in POF Cable when the cable or its pipe required repairs. A field trial wherein oil was frozen for 110 hours revealed that the steel pipe was not fractured by the mechanical stress of the repairs and the frozen oil was not melted by the heat from pipe welding.

It was determined that the total internal stress on the pipe during freezing and repairs was 19.67 Kg f/mm² (193 MPa). At 22 Kg f/mm² (216 MPa) a crack length of 100 mm was necessary to cause brittle fracture at -100°C, while at -175°C this crack length becomes approximately 20 mm. Consequently, temperatures close to -100°C were recommended for the procedure.

6.13 1976-1982 - Electricité de France¹⁰

A series of experiments was carried out on carbon steel and austenitic stainless steel pipes in diameters varying from 173 to 340 mm (7 to 13 in.). The tests revealed that thermal stresses generated by the cooling and heating process were insufficient to cause fracture of the piping.

It was concluded that it was possible to freeze pipe typically found in circuits of thermal or nuclear power plants with no detrimental effects.

6.14 1980-present - Imperial Oil, Sarnia, Ontario³⁰

Imperial Oil has used the technique on small-diameter, low-pressure cooling water lines and at least once on a vertical hydrogen line. In this case the hydrogen was displaced with water.

The lines are generally 150 to 200 mm (6 to 8 in.) in diameter and no material failures have resulted.

Difficulty in obtaining an ice plug was experienced in a branch line when hot water was flowing in the main line. This was alleviated by cooling the water.

Imperial has "backed-off" on the use of the technique following a cautionary report from Exxon Research.

Imperial Oil personnel have used dry ice themselves to freeze small-diameter lines.

6.15 1980-81 - Nova An Alberta Corporation, Calgary, Alberta³¹

Interested in using the process under license, Nova did a trial on a 150 mm (6 in.) diameter line. Experiencing no problems in the trial, Nova concluded that the technique was viable. However, it was decided that there was not a sufficient market in Canada to justify a licensing arrangement.

Nova cautioned that proper planning was necessary to ensure the procedure could be accomplished safely.

6.16 1980-81 - Suncor, Fort McMurray, Alberta³²

A procedure for freeze plugging was submitted to Suncor. It was examined by engineering personnel and was not approved due to concern of brittle fracture as the steel cools and heats.

6.17 1981 - Dow Chemical Company, Sarnia, Ontario³³

Dow Chemical Company experienced a cracked valve on a 250 mm (10 in.) service water line when the line froze under winter conditions. The flow of water was reduced as much as possible and a freeze was applied upstream of the valve.

The pipe was sealed off by an ice plug in approximately three hours, allowing replacement of the valve. No detrimental effects were noted on the pipe and the line operated for a number of years after the repair.

Plant management is somewhat reluctant to use the technique fearing potential failure.. However, under emergency situations the technique would be used again.

It was emphasized that proper operating procedures and precautions must be observed. These include freezing on a straight run of pipe far enough away from the repair area to ensure that the frozen section will not be subjected to stresses.

Dow personnel have done some freezing themselves on small-diameter piping using dry ice.

6.18 1980-81 - Polysar Corporation, Sarnia, Ontario³⁴

Freeze plugging was used on one occasion on a demonstration basis. A 200 mm (8 in.) diameter carbon steel line was frozen. Polysar personnel were not satisfied with the results, claiming they had no confidence that an ice plug had formed simply because ice was present on the outside of the pipe.

The technique was not approved for use since Polysar believed there was insufficient data to support claims of no damage to piping materials.

Polysar personnel vaguely remember the work that was done in the early 1970's but the records have been destroyed.

6.19 1981 - Experiments at Southampton University²³

Pipe freezing was studied in a 10 mm (4 in.) diameter carbon steel pipe. The study revealed that ice pressures were low in comparison to thermal stresses generated by the contraction of the piping material.

6.20 1980 - Trials Conducted by BCB Pipefreezing Services Ltd., by David Buckman³⁵

A laboratory trial on a 200 mm (8 in.) diameter, ebonite-lined pipe revealed that freezing did not damage the lining material.

In addition, a field trial on a 300 mm (12 in.) diameter PVC lined pipe, was reported. No damage to the liner occurred.

6.21 1982 - Battelle Columbus Laboratories¹⁴

Freezes were carried out on a 300 mm (12 in.) nominal diameter X52 pipe material. Freezes were conducted on sections both with and without defects. Defects were in the form of machined notches containing fatigue-induced cracks. No failures were experienced even though the stresses were such that both ductile and brittle fracture in the respective tests were expected. No pipe damage was revealed as a result of the freezing operation.

6.22 Canadian Insurance Industry

Several major insurance companies in Canada were contacted to obtain their reaction to ice plugging. Although there was some diversity of opinion, most indicated that ice plugging was not acceptable. In the event of an accident, loss would not be covered because the line or system was being subjected to conditions for which it was not designed.

Most companies had experienced claims due to the rupture of piping when lines froze under winter conditions. This rupture was normally accompanied by swelling of the line at other locations. Normally the swollen pipe was replaced because of concerns over stress corrosion cracking of the deformed material.

Most companies claimed the ductile/brittle transition experienced by carbon steels during ice plugging would give them concern. Should rupture occur, damage could be considerable.

Some companies indicated they would be prepared to accept an ice plug operation provided it was demonstrated through acceptable case histories and metallurgical studies that damage was unlikely. However, they stressed they would probably demand field trials on isolatable spool pieces before final approval would be given.

6.23 1984 - Exxon Company - Benicia Refinery⁴³

The bonnet bolts on a suction valve failed during an attempt to form an ice plug in the valve body to allow removal of a pump for repair. The freezing box had been built over the bonnet. During the freeze the bonnet bolts were overstressed and fractured.

6.24 1984 - Exxon Company - Baton Rouge⁴³

A "near-miss" hydrocarbon release occurred during an ice plugging operation. A section of piping containing hydrocarbon was being isolated by freezing to permit a valve replacement. When the flanges were spread, oil and water sprayed out, indicating that the ice plug had not sealed the line.

6.25 Date Not Given - Buckeye Pipeline Company⁴³

The Buckeye Pipeline Company experienced a rupture in a 150 mm (6 in.) diameter pipeline during a pressure test. The crack initiated in the pipe at a dent caused by a back-hoe during excavation. The end of the freeze jacket had been placed over half of the dent.

7. DISCUSSION

As noted previously, the lack of published information and the seeming reluctance of individuals to discuss ice plug technique is perceived by many as an attempt to hide potential problems. Certainly the ductile-to-brittle transition in fracture mode of carbon steels with decreasing temperature is well known and it is this behaviour that is the root of most of the uneasiness concerning ice plugging.

7.1 Ductile/Brittle Transition

Ductile-to-brittle transition is displayed graphically by depicting the amount of impact energy absorbed by a notched specimen (Charpy V-notch) at varying temperatures (Figure 1). The results of impact tests are interpreted on the basis of fracture appearance: ductile and brittle. Above the transition range the fracture appearance is 100% ductile (or shear) while below it the fracture appearance is brittle (cleavage). As transition ranges are typically in the 0° to 20°C range for the standard ferritic alloys used as piping materials, it is obvious that at liquid nitrogen temperatures the fracture mode would be completely brittle and would require very little impact energy to cause crack propagation.

A ductile to brittle transition in fracture mode occurs as well under static tensile conditions. However, unlike the impact conditions this transition occurs only at very low temperatures^{14,24}, generally below -100°C. This is attributed to a slow strain rate and the absence of notch effects. At the same time, both the yield strength and ultimate tensile strength increase; this increase may inhibit the material's ability to absorb a strain without fracturing.

Consequently, conditions arise in which the material requires a much higher tensile load to cause fracture but because of its inability to redistribute the stresses around notches at the lower temperature, fracture may occur. It is the lack of information about the response of defects in the piping material to the freeze conditions that is of grave concern.

In a series of tests performed by Battelle Columbus Laboratories on line-pipe steels it was demonstrated that fracture models were not completely adequate to predict pipe behaviour under ice plugging conditions^{14,24}. Although some of the test samples were expected to fail because the stress levels for both ductile and brittle failure were exceeded, they did not. This suggests that not only is the ductile failure criterion conservative, but the brittle one appears to be as well. Suggestions have been made that one or more of the following may be applicable¹⁴.

- (1) The analytical model for predicting brittle fracture is extremely conservative for surface defects in pipe under pressure.
- (2) The transition to brittle fracture initiation is not accompanied by a reduction in failure stress.
- (3) The stress level in the plug region is not as high as that in the rest of the pipe.

- (4) When the defects are taken to a high stress level and then cooled to a low temperature they are, in effect, warm proof-loaded.
- (5) The combination of low temperature and high stress never occurs without prior warm proof-loading.

While these tests suggest that the fracture of typical pipe steels during ice plugging is unlikely provided the material is not subjected to abnormal loading, the tested steel did not represent the lowest possible toughness. Therefore, generalization for all ice plug operations based on these tests, is impossible.

Similarly, it was indicated that the highest stresses and strains during ice plugging occurred at the inside surface on the top of the pipe but some defects tested were on the outside surface. Relocation of more defects to the highly stressed regions might have affected the experimental results.

In the Sarnia tests, defects on the outside of the pipe spools had little effect on the brittle fracture aspects of the ferritic steels. It was the defects at depth, more precisely in the root of the welds or adjacent heat affected zones, that were associated with failures; a point that is confirmed by the location of failures in pipelines that have been frozen. In this situation, fracture is most often associated with corrosion along the edge of a weld or at locations randomly distributed around the pipe circumference where large corrosion pits exist⁴¹.

In the absence of notch effects, the pipe may swell but rupture has not occurred as the stress was redistributed.

Another factor in controlling brittle fracture during ice plugging is suggested by the observation that the plug region, which would be subjected to the lowest temperatures, was isolated by the plug from any pressure effect in the pipe. The area of isolation extended to within 100 mm of the plug ends. Consequently, any thrust effect due to pressure in the pipe was quickly absorbed by the plug. Thus it would appear that thermally-induced stresses constitute the greatest threat of fracture.

indeed, experiments confirm that the thermally-induced stresses, although complex, are considerably lower than the yield stress of the pipe, particularly in the absence of internal pressure^{11,14}. With internal pressure, the thermally-induced stresses are much higher on the inside surface than on the outside surface, at the top of the pipe. The stress is tensile on the inside surface and compressive on the outside surface as the pipe ovals, suggesting that defects on the inside of the pipe might be more likely to propagate than those on the outside surface¹⁴.

The highest stresses found, although significant, are still below those required to propagate cracks unless the cracks are already relatively large. These high stresses occur during the maximum thermal gradient at the start of cooling when the pipe is still warm, retaining its toughness. The stresses are reduced significantly once the ice plug forms.

Critical crack sizes may be calculated from various formulae. These formulae assume a semi-elliptical crack shape, which is not severe when compared with sharp defects produced by corrosion, fatigue or mechanical stresses. Nonetheless, the response to brittle fracture has been shown to be conservative, so the crack size determinations appear reasonable¹⁴.

Critical crack depth, or critical crack length permitting propagation, decreases with decreasing temperature. At liquid nitrogen temperatures, the critical crack depth could approximate one to ten millimetres, depending on stress levels¹¹. A 1 mm deep crack could be overlooked with standard Non-Destructive Testing procedures. Indeed, even larger defects could be missed as interpretation depends on the skill of the operator. Consequently, reliance on a single non-destructive evaluation technique to ensure freedom from injurious defects may be impractical. However, it will permit detection of gross defects that certainly will propagate during ice plugging and, as such, is an effective method of contributing to a safe procedure.

7.2 Plug Slippage

Plug slippage is an obvious hazard that has been reported, albeit verbally, during ice plugging. The slippage that has occurred is usually the result of premature removal of the cooling medium, either intentionally to reduce costs or accidentally because of insufficient coolant.

As long as a freeze is maintained properly there is ample evidence that a plug will not slip even when subjected to relatively high pressures.

Retention of an ice plug is attributed, in part, to the "hour glass" shape of the pipe (suggesting a minimal radial stress is exerted on the pipe by the ice) and to a bond established between the ice the steel¹⁴. This bond effectively absorbs the thrust energy over a very short distance so that the majority of the ice plug would be isolated from any slipping tendency.

Judicial placement of thermocouples around the pipe during the freezing operation would permit the recording of temperature variations. As long as the ice plug is in place and the low temperatures remain constant, slippage is unlikely¹⁴.

7.3 Restraint

As a section of piping is cooled, the piping material attempts to contract longitudinally, inducing a longitudinal stress in the pipe. The amount of stress depends on the length of pipe cooled and on the location of the pipe restraint. If the pipe is restrained immediately adjacent to the ice plug area, it will experience a very high stress and fracture may occur. It is obvious that the longer the span of unrestrained pipe, the lower the maximum longitudinal stress imposed by the cooling.

Since it is relatively easy to extend the span of unrestrained pipe, the longitudinal stress would be significant only in the presence of a circumferentially-oriented defect. The most common place for such defects is

in circumferential welds. Consequently, the restraint problem can best be avoided by not freezing in the vicinity of circumferential welds.

7.4 Defects

Piping systems are rarely completely free of defects but it has been demonstrated that some defects can be tolerated in the most severely stressed regions of a pipe during ice plugging and still not cause failure¹⁴. Therefore, it seems unreasonable to expect that areas of pipe to be frozen should be completely free of defects. They should, however, be as defect-free as is practically possible.

Most commercial pipe mills use some form of ultrasonic shear wave inspection to detect defects in their products. These methods are relatively accurate for thick-wall piping because the probability of locating crack-like defects penetrating over 10 percent of the wall thickness is 100%. Even in thinner material the probability of finding defects approaching 50% or more of the wall thickness is very high. Since defects approaching 50% of the wall thickness did not cause failure under ice plug conditions^{14,24}, it seems reasonable that critical defects in ice plug locations could be located using standard ultrasonic shear wave technique.

Defects in piping may be the result of a number of processes involving manufacturing, corrosion, fatigue, and mechanical damage. The most common defects are usually associated with welding and occur not only in the weld metal but also in the adjacent parent metal (heat-affected zone).

Tests have revealed that certain weld defects present more problems than others^{13,14,42}. Any form of sharp surface defect is more likely to promote fracture than an internal defect. Consequently, cracks, incomplete penetration, incomplete fusion, centre-line crevices and undercut in welds would be expected to promote brittle fracture under ice plugging conditions and this has been confirmed¹³.

As well, any form of sharp surface defect such as that produced by corrosion, stress corrosion or fatigue would be expected to promote brittle fracture. Such defects have been found to create problems with ice plugging^{13,14,24}.

Finally, gouges, dents and scratches produced by mechanical damage may be considered as hazardous. These features can mask more serious cracking and have been responsible for at least one pipeline failure during a freezing operation.^{14,43}

Using standard inspection techniques it is possible to identify pipe areas containing circumferential welds, fittings, repair welds, cracks, large pits, excessive corrosion, dents, gouges, or leaking defects and to exclude these as possible sites for an ice plugging operation.

8. RECOMMENDATIONS FROM COMPANIES USING ICE PLUGGING

While there have been failures during ice plugging operations, a number of companies consider the technique a valuable hot tapping procedure and have outlined recommendations to help prevent fracture, particularly with ferritic alloys. They caution, however, that failure to follow the recommendations could result in serious accidents and/or the release of large quantities of hazardous or valuable product. The recommendations are summarized below.

- (1) Each application of freeze plugging should be preceded by a risk analysis including the development of an emergency plan should rupture occur.
- (2) All lines should be depressurized before attempting a freeze.
- (3) Freeze plugging should be used as an emergency measure, not as a standard maintenance procedure.
- (4) The area to be frozen must be inspected before and after freezing both visually and ultrasonically using a shear wave technique. However, it is cautioned that defects which might propagate to failure could still be missed.
- (5) Freezes should not be permitted on girth welds, on repair welds, on fittings, on complicated piping configurations, nor on areas containing evidence of heavy corrosion.
- (6) Venting during freezing is necessary to prevent rupture of the pipe, particularly if the freeze occurs near the end of a run or near a change in direction.
- (7) Freezes should be avoided on cold-worked material.
- (8) Hydrocarbons should not be frozen.
- (9) Freezing should take place only on straight runs of pipe, maintaining a minimum length of ten pipe diameters from the end of the freeze area to a point of restraint.
- (10) Adequate spacing must be maintained between two plugs, with a minimum of twenty pipe diameters suggested. If cutting or welding is to take place between the plugs, this distance should be increased to maintain ten pipe diameters from the heat-affected area to the edge of each freeze location.
- (11) The freeze jacket should not be removed to permit a rapid thaw after pipe repairs.
- (12) Each pipe freeze should be treated as a significant event and recorded as such.
- (13) Excessive stresses on the pipe must be avoided while the freeze is in progress. Such features as torque, impact, or improper

support of a line once a section has been removed, may lead to failure.

- (14) The presence of frost on the pipe is not a definite indication that an ice plug has formed; the plug integrity should be tested hydrostatically or a positive sign such as the stoppage of a visible leak should be obtained before cutting into the pipe. Thermocouples should be used to record the pipe temperature.
- (15) Personnel should approach the freeze area only when necessary during the freeze.
- (16) Carbon dioxide rather than liquid nitrogen should be used as a freezing medium to reduce thermal gradients and lower stresses.

In addition to general recommendations which govern most freeze operations, Electricité de France has refined its technique, particularly as it applies to nuclear facilities³⁹. Freezing is prohibited on carbon steel alloys, particularly in primary circuits, confining the technique to austenitic stainless steels, types 304 and 316. A freeze is permitted only after a complete safety analysis of the event has been performed.

9. CONCLUSIONS

This study has established that ice plugging in Canada as well as elsewhere is not widespread outside the nuclear industry. Approximately half of major Canadian companies have used the technique only once on relatively small diameter lines (150 to 200 mm) and they suggest its use was in response to a vigorous marketing campaign conducted by commercial freezing companies between 1978 and 1982. The practice appears to be declining in popularity due to concerns about brittle fracture of ferritic alloys, concerns over insurance coverage, and fears raised by accidents, both real or rumoured, in Europe and North America. Even ardent supporters of the technique advise extreme caution in its application.

Ice plugging does appear to be a viable method of sealing pipe in non-hazardous service provided proper planning and adherence to very specific procedures without deviation is observed. Nevertheless, surface defects can lead to brittle fracture, particularly in ferritic alloys. Defects that may cause problems are those located in areas of high stress and generally are sharp in profile (such as cracks, excessive corrosion and linear weld defects). Judicial choice of a freeze location may eliminate some of these problems.

Some work has been done to assess the metallurgical response of ferritic alloys to the freeze process but more research is needed to fully address the concerns of brittle fracture due to notch considerations of defects. Most reports to date involve studies on relatively small diameter seamless piping and are not totally applicable to welded material.

Ice plugging appears suited for use on piping material up to 300 mm in diameter. Above this size, there is insufficient data available to justify its application.

Finally, it appears that some industries, oil and gas in particular, think that the use of liquid nitrogen as a coolant increases the risk of brittle fracture in ferritic alloys due to higher thermal gradients. Freeze plugging with carbon dioxide is advocated to reduce both stresses and the associated risk of brittle fracture.

10. RECOMMENDATIONS

Based on this study there appears to be no reason to avoid the practice of ice plugging provided it is conducted with caution using proper procedures. In addition to the recommendations outlined by ice plug users, the following should help to ensure a safe operation.

- (1) The use of ice plugging should be limited to piping with a maximum diameter of 300 mm until technical data is produced to justify exceeding this size.
- (2) Repeated freezes should be avoided in the same location on ferritic alloys to prevent a potential decrease in steel toughness.
- (3) Homemade freezing devices should be avoided to prevent accidents.
- (4) Each freezing operation should be treated as a significant event with a report issued on the occurrence.
- (5) Considerably more research is required on the response to freezing of pipe defects, particularly those associated with welding, in order to approximate more closely field conditions.
- (6) Work is required on the liquid nitrogen versus carbon dioxide aspects of preventing fractures by lowering thermal gradients particularly when defects exist in highly stressed areas.

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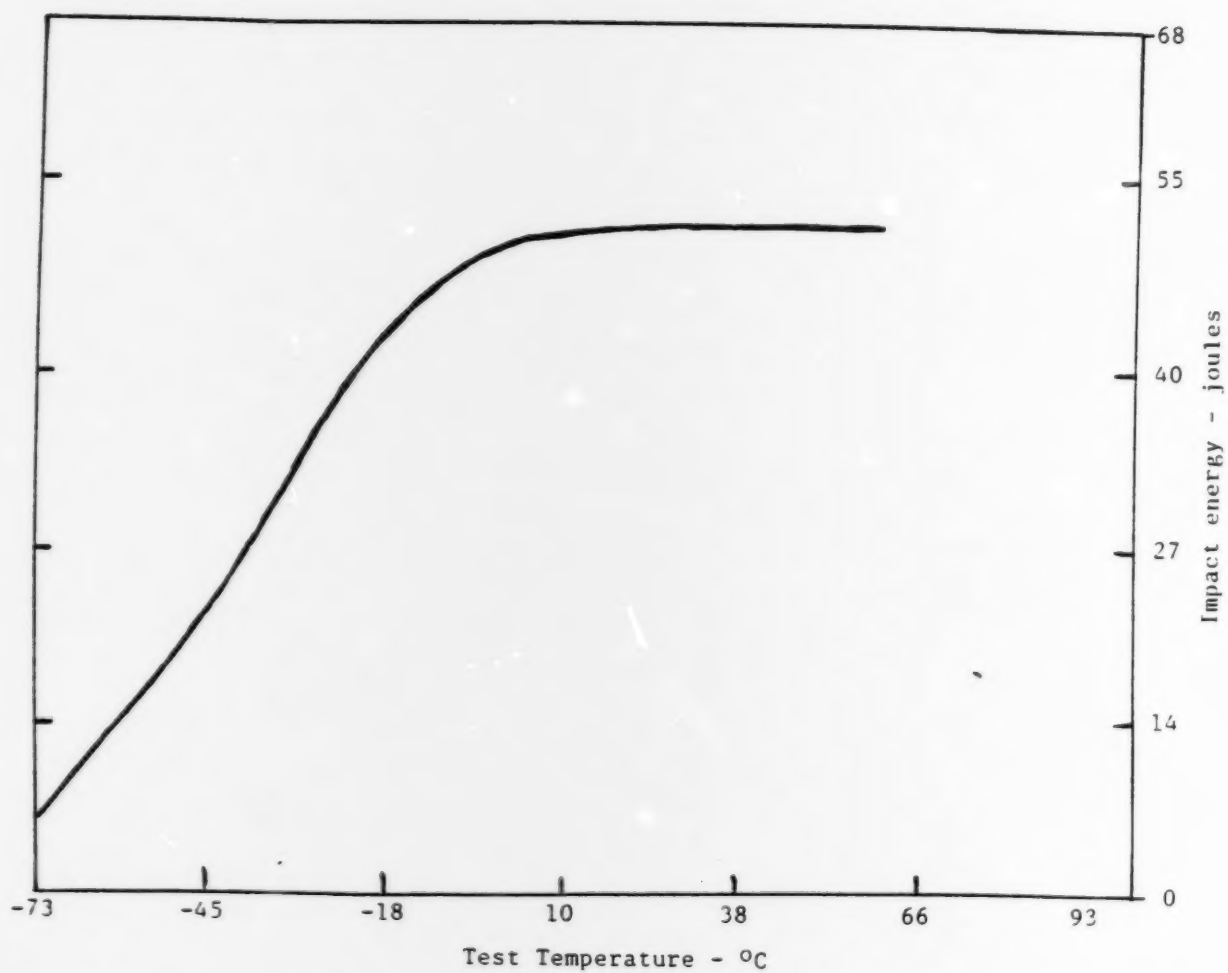


Figure 1: X 65 Charpy V-Notch Impact Test Results

(After J.F. Kiefer et al, Reference 14)